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RESEARCH ARTICLE

The Effect of Immediate Dentin Sealing Using Different Universal Adhesives on the Bond Strength of Pretreated Monolithic Zirconia to Dentin and Microscopic Morphological Alterations

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ABSTRACT

This study evaluated the effects of immediate dentin sealing (IDS) using different universal adhesives on microtensile bond strength (μTBS) of pretreated monolithic translucent tetragonal polycrystalline zirconia-based CAD/CAM restoration to dentin and microscopic morphological alterations. Mid-coronal dentin were obtained from 32 human molars and randomly allocated into 8 groups according to the presence/absence of IDS, universal adhesives for IDS (Single Bond Universal/SBU, Clearfil Quick Bond/CUQ, Optibond Universal/OBU) and zirconia surface pretreatments (tribochemical silica coating-30 μm CoJet particles/TSC, sandblasting-50 μm Al_2O_3/SB): Group TSC, Group TSC+SBU, Group TSC+CUQ, Group TSC+OBU, Group SB, Group SB+SBU, Group SB+CUQ, Group SB+OBU. CAD/CAM restorations (Lava Plus High Translucency Zirconia Disc) were produced, luted, and subjected to 10.000 thermocycles (5°C–55°C). Zirconia–dentin bars (2 × 2 × 8 mm) were subjected to μTBS test ($n = 16$). Surface morphology of zirconia and zirconia–dentin interfaces were analyzed under SEM ($n = 1$). Two-way ANOVA and Bonferroni tests were used for statistical analyses ($p < 0.05$). Regarding the presence of IDS, Group TSC + OBU showed statistically higher μTBS than Group TSC ($p < 0.05$). Comparing the IDS applied groups, Group TSC+OBU and Group SB+OBU showed statistically higher μTBS than Group TSC+CUQ and Group SB+CUQ, respectively ($p < 0.05$). According to the surface pretreatments, no significant differences in μTBS were found ($p > 0.05$). A thick, intact hybrid layer with long resin tags were observed for Group TSC+OBU and Group SB+OBU. After tribochemical silica coating or sandblasting pretreatments of monolithic zirconia, IDS procedure with Optibond Universal could affect the bond strength to dentin and morphological appearance, positively.

1 | Introduction

Monolithic ceramic restorations have facilitated a more uniform structure, preventing porcelain veneer chipping and delamination from the core when processed via computer-aided design and computer-aided manufacturing (CAD/CAM) systems (Tabatabaian, Karimi, and Namdari 2020). Highly translucent CAD/CAM zirconia has been widely used for monolithic restorations due to their natural tooth color, translucency, optimal biocompatibility, suitable marginal fit, and satisfactory

Summary

- Within the limitations of this in-vitro study, it can be concluded that:
  - After TSC or SB pretreatments of monolithic zirconia, IDS procedure with OBU could affect the μTBS to dentin and morphological alternations, positively.
  - SEM images of OBU revealed that thick hybrid layer with longer resin tags was observed.

wear of the antagonist (Sen and Isler 2020). LavaTM Plus High Translucency Zirconia (3M ESPE, USA) is a tetragonal polycrystalline zirconia partially stabilized with 3 mol% yttria engineered for high translucency and utmost strength (Albakry, Guazzato, and Swain 2004).

Successful adhesion between ceramic-based materials and resin cement is required. Thus, the strength of both tooth and indirect restoration, retention, marginal adaptation, and sealing of indirect restorations are enhanced. Therefore, the surface treatments are required to provide a proper adhesion between resin cement and ceramic (Spitznagel et al. 2014).

Traditional silica-based ceramics can possess satisfactory bond strengths through a combination of chemical adhesion and micro-mechanical retention. Unfortunately, high crystalline ceramics, including silica-free monolithic zirconia, were previously considered incapable of comparable bonding (Thompson et al. 2011). To overcome this problem, it was necessary to roughen the cementation surface so that surface energy, surface wettability and micromechanical interlocking would increase (Abi-Rached et al. 2014). Various surface treatments have been proposed, including sandblasting with aluminum oxide (Al_2O_3), tribochemical silica coating, laser irradiation, selective infiltration etching, and chemical etching (Tzanakakis, Tzoutzas, and Koidis 2016).

Immediate dentin sealing (IDS) application is another method that could provide the improved bond strength of indirect restorations (Magne et al. 2005). That procedure involves an adhesive system or/and flowable composite resin application instantly after tooth preparation and before impression taking.

It has been stated that IDS could protect freshly prepared dentin from contamination and preserve the structure of the tooth. It also improves patient comfort during the provisional restoration phase and reduces the need for anesthesia during the cementing appointment for the definitive restoration (Rigos et al. 2019). Besides, the hybrid layer remains unaffected by the tensile stresses applied during cementation, thus protecting the collagen fibrils within it from breaking down. Hence, the luting procedure of indirect restorations lead to higher bond strength (van den Breemer et al. 2019).

Etch&rinse adhesive systems have traditionally been recommended for the IDS applications. However, the effectiveness of self-etch adhesive systems has been reported that those simplified systems in IDS applications could also increase bond strength (Pheerarangsikul, Wayakanon, and Wayakanon 2022). There is a trend in adhesive dentistry to simplify the adhesion steps to reduce the time spent in the chair and the technical precision of clinical procedures. In the recent years, new one-step self-etch adhesive systems, known as universal adhesive systems, have been introduced to the market. These adhesive systems can be bonded to different dental hard tissues and restorative materials. In addition, "multimode" option can be applied as self-etch, etch&rinse, and selective-etch modes (Pires et al. 2019).

In the literature, there is not enough information about the adhesion of monolithic translucent zirconia blocks to dentin after the use of different universal adhesive systems for the IDS application. In addition, it is difficult to provide a gold standard protocol on the surface pretreatment methods before the cementation of zirconia-based ceramics. Therefore, the aim of the present study was to evaluate the effect of IDS application using different universal adhesive systems on microtensile bond strength (μTBS) of pretreated monolithic translucent tetragonal polycrystalline zirconia-based CAD/CAM restoration to dentin and microscopic morphological alterations.

The null hypothesis of this study was that the IDS application using different universal adhesive systems would not affect the μTBS of pretreated monolithic translucent tetragonal polycrystalline zirconia-based CAD/CAM restoration to dentin and microscopic morphological alterations.

2 | Materials and Methods

This in vitro study has been approved by a local ethics committee (Process number: 2021/252).

The specimen size was stated on the basis of the expected effect size between the two groups, as described in the relevant literature (Gailani et al. 2021). For the microtensile bond strength test, the specimen size required for per group to acquire a medium effect size ($d = 0.50$) with 80% power and a type 1 error rate of 5% was 16.

2.1 | Specimen Preparation

Totally, thirty-two extracted sound human lower molars were collected. Twenty-four teeth were used for microtensile bond strength test (μTBS) and eight teeth were used for morphological examination of the dentin–zirconia interface with scanning electron microscope (SEM). They were kept in a saline solution until the experimental procedures were carried out at room temperature and used within 3 months of extraction. A diagram of the experimental procedure is given in Figure 1. The restorative materials used in this study, their brand names, manufacturers, batch numbers, and chemical compositions are shown in Table 1.

The occlusal 1/3 of the crown was removed using a slow-speed diamond saw with a model trimmer (MT3 Wet trimmer, Renfert GmbH; Hilzingen, Germany) under water cooling to fully expose the dentin. The flat middle-depth coronal dentin was exposed and then polished with 600-grit silicon carbide paper on a

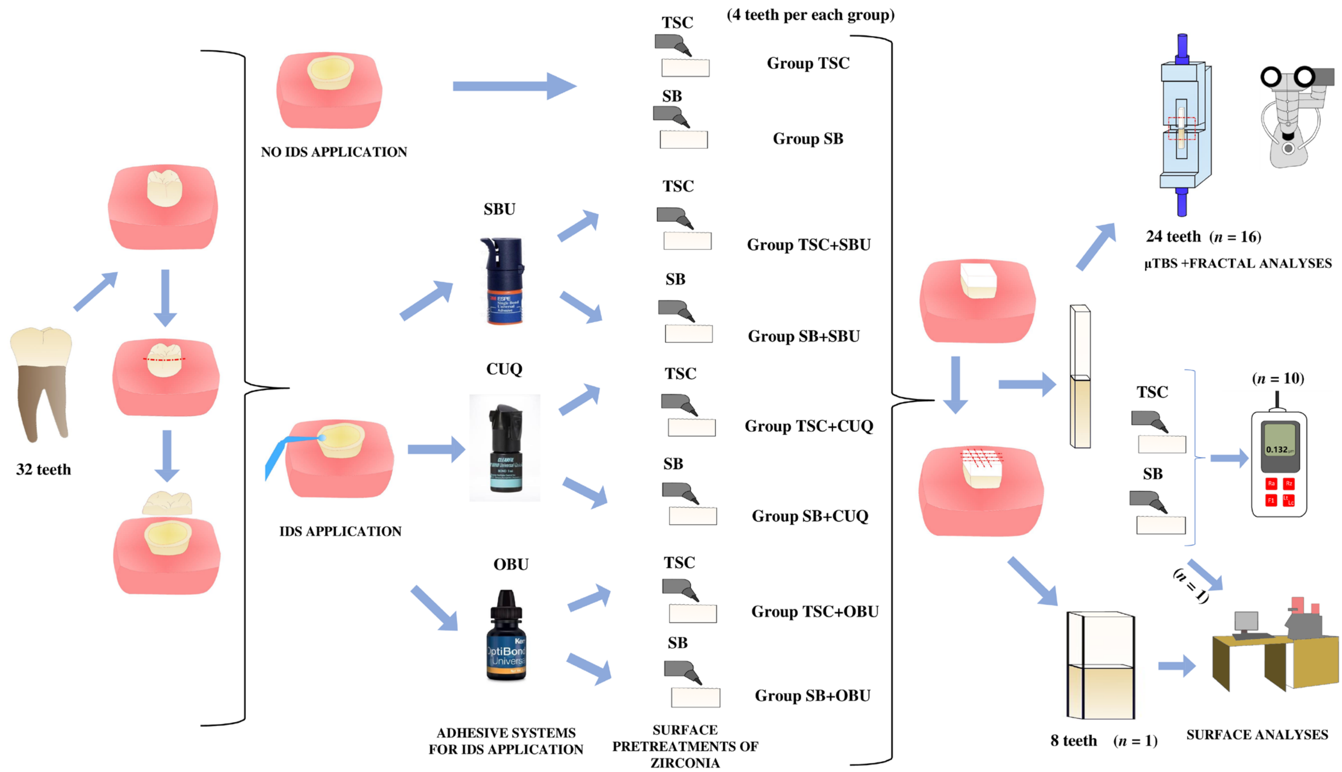

FIGURE 1 | Diagram of the experimental procedure.

TABLE 1 | The brand names, manufacturers, batch numbers, and chemical composition of restorative materials used in this study.

	Brand names	Manufacturer	Batch number	Composition
Adhesive systems	Single Bond Universal	3 M ESPE; USA	8220153	10-MDP phosphate monomer, HEMA, dimetalrylate resin, vitreous bond copolymer, ethanol, water, silane (pH: 2.7)
Adhesive systems	Clearfil Universal Bond Quick	Kuraray, Japan	BH0329	10-MDP, Bis-GMA (10%–25%), HEMA (2.5%–10%), hydrophilic amide monomers, colloidal silica, silane, Sodium fluoride, camphoroquinone, ethanol, (10%–25%) water (pH: 2.3)
Adhesive systems	Optibond Universal	Kerr, Orange, USA	7833990	Aceton (30%–6%0), HEMA (5%–10%), GPDM 1–5 (%), ethanol (5%–10%), water (pH: 1.9)
Resin cement	Rely X U2OO	3 M ESPE, USA	8354116	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives)
Zirconia based CAD/CAM block	Lava Plus High Translucency Zirconia Disc	3 M ESPE, USA	8797557	98S-18 mm disc tetragonal polycrystalline zirconia partially stabilized with 3 mol-% yttria, 0.1% alumina

Abbreviations: %, percentage; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate;BIS-GMA, bisphenol A-glycidyl methacrylate; GPDM, glycero-phosphate dimethacrylate; HEMA, hydroxylethyl methacrylate.

metallographic polisher (Minitech 233, Presi; Grenoble, France) under water cooling to create a proper surface standardization (Rigos et al. 2019). The specimens were examined under a stereomicroscope to detect the presence of enamel or pulp exposure (SMZ 1000, Nikon; Japan).

The specimens of 4 mm thickness, 10 mm width and 10 mm length were obtained from a monolithic translucent zirconia-based CAD/CAM block (Lava Plus High Translucency Zirconia Disc, 3M ESPE, USA) using a Lava Milling System (Coritec 550i, Imescore, Germany) (Gailani et al. 2021). Subsequently, the specimens were sintered according to the manufacturer's instructions.

Then, they were randomly allocated into eight groups according to the presence/absence of IDS application, the type of universal adhesive systems used for IDS application (Single Bond Universal, Clearfil Quick Bond, Optibond Universal) and surface pretreatment methods for the monolithic zirconia-based CAD/CAM blocks (tribochemical silica coating method/TSC and sandblasting/SB):

2.1.1 | Group TSC (Tribochemical Silica Coating+No IDS/Control)

The surfaces of CAD/CAM specimens were pretreated with tribochemical silica coating method using 30 µm silica-coated aluminum oxide (Al_2O_3) particles (CoJet Sand, 3M ESPE, USA) at 2.8 bar pressure with a distance of 10 mm for 10 s (Rigos et al. 2019). The residues were cleaned in an ultrasonic bath with isopropyl alcohol for 3 min and air-dried. The exposed dentin surfaces did not receive IDS treatment.

2.1.2 | Group TSC+SBU (Tribochemical Silica Coating+Single Bond Universal)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with tribochemical silica coating method. For IDS procedure, according to the manufacturer's instructions, Single Bond Universal (3M ESPE; St. Paul, MN, USA) adhesive system was utilized to the exposed dentin surfaces in self-etch mode using a microbrush for 20 s, gently air-thinned for 5 s and light-cured for 10 s (irradiance of 1000 mW/cm^2) with a light-emitting diode light-curing unit (LED LCU) (Valo, Ultradent; South Jordan, UT, USA).

2.1.3 | Group TSC+CUQ (Tribochemical Silica Coating+Clearfil Universal Bond Quick)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with a tribochemical silica coating method. For IDS procedure, according to the manufacturer's instructions, Clearfil Universal Bond Quick (Kuraray, Okayama, Japan) adhesive system was utilized to the exposed dentin surfaces in self-etch mode with a rubbing motion using a microbrush for 20 s, air-dried until the bond did not move, and light-cured for 10 s (irradiance of 1000 mW/cm^2) with LED LCU.

2.1.4 | Group TSC+OBU (Tribochemical Silica Coating+Optibond Universal)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with tribochemical silica coating method. For IDS procedure, according to the manufacturer's instructions, Optibond Universal (Kerr Corp., Orange CA, USA) adhesive system was utilized to the exposed dentin surfaces in self-etch mode using a microbrush for 20 s, gently air-thinned for 5 s, and light-cured for 10 s (irradiance of 1000 mW/cm^2) with LED LCU.

2.1.5 | Group SB (Sandblasting+No IDS/Control)

The surfaces of CAD/CAM specimens were pretreated with sandblasting method using 50 µm Al_2O_3 particles at 3 bar pressure with a distance of 10 mm for 10 s (Rigos et al. 2019). The residues were cleaned in an ultrasonic bath with isopropyl alcohol for 3 min and air-dried. The exposed dentin surfaces did not receive IDS treatment.

2.1.6 | Group SB+SBU (Sandblasting+Single Bond Universal)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with sandblasting method. For IDS procedure, as mentioned earlier, according to the manufacturer's instructions, Single Bond Universal (3M ESPE, St. Paul, MN, USA) adhesive system was utilized to the exposed dentin surfaces in self-etch mode and light-cured for 10 s (irradiance of 1000 mW/cm^2) with LED LCU.

2.1.7 | Group SB+CUQ (Sandblasting+Clearfil Universal Bond Quick)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with sandblasting methods. For IDS procedure, as mentioned earlier, according to the manufacturer's instructions, Clearfil Universal Bond Quick (Kuraray, Okayama, Japan) adhesive system was utilized to the exposed dentin surfaces in self-etch mode and light-cured for 10 s (irradiance of 1000 mW/cm^2) with LED LCU.

2.1.8 | Group SB+OBU (Sandblasting+Optibond Universal)

As mentioned above, the cementation surfaces of monolithic zirconia-based CAD/CAM blocks were pretreated with sandblasting method. For IDS procedure, as mentioned earlier, according to the manufacturer's instructions, Optibond Universal (3M ESPE; St. Paul, MN, USA) adhesive system was utilized to the exposed dentin surfaces in self-etch mode and light-cured for 10 s (irradiance of 1000 mW/cm^2) with LED LCU.

After IDS applications, the dentin specimens were kept at 37°C in distilled water, until the luting procedures.

2.2 | Luting Procedure

Rely X U200 (3M ESPE, USA), a self-adhesive dual-cure resin cement, was used for pretreated monolithic zirconia-based CAD/CAM blocks luting. The base and catalyst of the resin cement were mixed for 20 s on the mixing pad according to the manufacturer's instructions. After the application of cement, the pretreated surfaces of monolithic zirconia-based CAD/CAM blocks were positioned on the dentin surfaces. A constant load of 50 N was then applied throughout the setting time of the cement (Pilo et al. 2018). Overflowing cement residues were removed with a cotton pellet. The cemented blocks were polymerized from 5 different surfaces (occlusal, buccal, lingual, mesial, and distal) with an LED LCU (1000 mW/cm^2) for 20 s.

A radiometer (Demetron LED Radiometer, Kerr Corp., CA) was utilized throughout the procedure to check the radiant energy (1000 mW/cm^2). All restorative procedures were conducted by one operator following the manufacturer's instructions (L.F.). Then, all specimens were kept in distilled water 37°C for 24 h.

Then, the specimens were submitted to 10.000 thermocycles (5°C–55°C, dwell time:30 s and transfer time:10 s) with a thermocycle device (SD Mechatronic Thermocycler, Germany).

2.3 | Microtensile Bond Strength Test (μTBS)

Twenty-four teeth were used for μTBS test. For study groups as mentioned above, specimens were vertically sectioned to obtain zirconia–dentin bars (2 × 2 × 8 mm) with a slow-speed diamond saw of a micro-cutting device (Isomet 1000, Buehler Ltd. Lake Bluff IL, USA) under water cooling. The bars were examined under a stereomicroscope and structurally intact and crack-free bars were selected. A total of 128 zirconia–dentin bars were used for eight groups ($n = 16$). Each specimen size was verified by measuring with a digital caliper. Then, they were fixed with cyanoacrylate gel to a unitary gripping device and submitted to μTBS test with a universal test machine (Microtensile Tester, Bisco Inc. USA) until failure (crosshead speed:0.5 mm/min). The μTBS was recorded as megapascal (MPa) as the maximum loading force (N) divided by the bond area (mm^2).

2.4 | Failure Mode Analysis

After μTBS test, fracture patterns of specimens were analyzed using a stereomicroscope (Leica MZ 21, Leica Microsystems, Turkey) under 15× magnification. The type of failure was recorded and the classification of the failure was as follows:

- Adhesive failure in zirconia and luting cement (less than one-third of the debonded zirconia surface presented luting cement residues).
- Adhesive failure in dentin and luting cement (less than one-third of the debonded dentin surface presented luting agent residues).
- Cohesive failure (two-thirds of the debonded surface presented luting cement residues on both the zirconia surface and the dentin).
- Mixed failure (adhesive and cohesive).

2.5 | Surface Roughness Analysis by Contact Profilometer

To evaluate the surface roughness values after surface pretreatments of monolithic zirconia, CAD/CAM blocks were vertically sectioned to obtain zirconia specimens (2 × 2 × 2 mm) with a slow-speed diamond saw of a micro-cutting device (Isomet 1000, Buehler Ltd., Lake Bluff IL, USA) under water cooling. Tribochemical silica coating and sandblasting surface pretreatments were applied to CAD/CAM specimens as mentioned above. Then, surface roughness (Ra, μm) of totally 20 CAD/CAM specimens were measured with a contact profilometer (Mahr GmbH, Mahrsurf PS1, Göttingen, Germany) ($n = 10$). Three measurements were taken at various points of the polished surface for each specimen, with following settings; stylus tip radius:5 μm, a stylus driving speed: 0.5 mm/s, traversing length (Lt): 1.75 mm, and five cut-off lengths: 0.25 mm. The mean values of the measurements were calculated.

2.6 | Surface Roughness Analysis by Scanning Electron Microscope (SEM)

After surface roughness measurements of monolithic zirconia, one representative CAD/CAM specimen (2 × 2 × 2 mm) from each pretreatment methods was analyzed with SEM (Evo LS10, Zeiss, Oberkochen, Germany) to evaluate the surface morphology ($n = 1$). The specimens underwent gold-sputter coating under secondary mode at an accelerating voltage ranging from 10 to 30 kV. The images of representative surfaces were examined at magnifications of 1000× and 2000×.

2.7 | Zirconia–Dentin Interface Analysis by Scanning Electron Microscope

Eight teeth were used for the ultrastructural morphological analysis of the zirconia–dentin interfaces with SEM. Representative specimens from each group were prepared as mentioned above for study groups. The cementation of pretreated monolithic zirconia-based CAD/CAM blocks to dentin were completed according to the earlier experimental protocol. Then, they were stored in distilled water at 37°C for 24 h. The bonded interfaces were obtained as cross-sectioned on the sagittal plane with a micro-cutting device under water cooling. Then, dentin was demineralized by submerging it in 6 N HCL for 30 s, followed by a rinse of water for 5 min. They were immersed in a 1% solution of NaOCl for 10 min, followed by a rinse with water for 5 min for deproteinization (Perdigão, Lopes, and Gomes 2008).

After drying at room temperature, the specimens underwent gold-sputter coating under secondary mode at an accelerating voltage ranging from 10 to 30 kV. The images of representative surfaces were examined at magnifications of 1.000×, 2.000×, and 5000×.

2.8 | Statistical Analysis

The statistical analysis was carried out using SPSS 22.0 for Windows (SPSS Inc., Chicago, IL). To assess the normality of variables, the Shapiro–Wilk test was conducted initially, followed by an analysis of homogeneity of variances using Levene's test. The data were found to be normally distributed. A two-way analysis of variance has been carried out to compare differences within and between groups. All pairwise comparisons were carried out with the Bonferroni test. For all analyses, a confidence level of 0.05 was determined for statistical significance.

3 | Results

3.1 | Microtensile Bond Strength Test

Table 2 exhibits the mean μTBS values with standard deviations (±SDs) in MPa for all tested groups.

Regarding the presence of IDS, Group TSC+OBU showed statistically higher μTBS than Group TSC ($p < 0.001$). However, Group SB+SBU, Group SB+CUQ, and Group SB+OBU showed similar μTBS to Group SB ($p = 0.015$).

When comparing the IDS applied groups; Group TSC+OBU showed statistically higher μTBS than Group TSC+CUQ ($p < 0.001$), whereas Group SB+OBU exhibited statistically higher μTBS than Group SB+CUQ ($p = 0.015$).

Regarding the surface pretreatment methods, no significant differences in μTBS were observed among all tested groups ($p > 0.05$).

TABLE 2 | The mean μTBS values and standard deviations (±SDs) of all tested groups in MPa ($n = 16$).

Groups	TSC	SB	*p*
IDS(−) (control)	1.984 ± 0.674^{a}	2.206 ± 0.629^{ab}	0.418
SBU	2.563 ± 0.851^{ab}	2.264 ± 0.977^{ab}	0.277
CUQ	2.344 ± 0.778^{a}	2.079 ± 0.771^{a}	0.334
OBU	$3.287 \pm 0,887^{b}$	2.903 ± 0.515^{b}	0.162
p	< 0.001	0.015	

Note: Different superscript small letters indicate the significant differences within the same columns ($p < 0.05$).
Abbreviations: Group SB, sandblasting+no IDS; Group SB+CUQ, sandblasting+Clearfil Universal Bond Quick; Group SB+OBU, sandblasting+Optibond Universal; Group SB+SBU, sandblasting+Single Bond Universal; Group TSC, tribochemical silica coating+No IDS; Group TSC+OBU, tribochemical silica coating+Optibond Universal; Group TSC+CUQ, tribochemical silica coating+Clearfil Universal Bond Quick; Group TSC+SBU, tribochemical silica coating+Single Bond Universal.

TABLE 3 | The mean surface roughness values (Ra, μm) values and standard deviations (±SDs) of all tested groups ($n = 10$). ($p < 0.05$).

Surface pretreatment methods	Surface roughness (±SD)
TSC	0.414 ± 0.153
SB	0.326 ± 0.126
p	0.177

Abbreviations: SB, sandblasting; TSC, tribochemical silica coating.

3.2 | Surface Roughness Analysis by Contact Profilometer

Table 3 presents the mean surface roughness values of monolithic zirconia-based CAD/CAM blocks with standard deviations (±SDs) after surface pretreatments for all tested groups.

According to the contact profilometry measurements, no significant differences in surface roughness were found among the pretreatment methods ($p > 0.05$).

3.3 | Surface Roughness Analysis by Scanning Electron Microscope

Representative SEM images of monolithic zirconia-based CAD/CAM blocks after surface pretreatments are shown in Figure 2. According to SEM analysis of surface morphology, specimen pretreated with TSC exhibited a rougher and irregular appearance (Figure 2a) while fewer rougher areas were observed for the specimen pretreated with SB (Figure 2b).

3.4 | Zirconia–Dentin Interface Analysis by Scanning Electron Microscope

Representative SEM images of the zirconia–dentin interfaces are shown in Figure 3. When the groups with no IDS application were compared, gap formations in the hybrid layer in some regions were found for Group TSC (Figure 3a,A); whereas a thicker and more continuous hybrid layer was detected for Group SB (Figure 3e,E). When the IDS applied groups were compared, a thick and intact hybrid layer with long resin tag formations were observed for Group TSC+OBU (Figure 3d,D) and Group SB+OBU (Figure 3h,H). Group TSC+SBU (Figure 3b,B), Group SB+SBU (Figure 3f,F), Group TSC+CUQ (Figure 3c,C), and Group SB+CUQ (Figure 3g,G) exhibited a thin and continuous hybrid layer with short resin tags in some areas.

3.5 | Failure Mode Analysis

Table 4 displays the failure modes of fractured surfaces after the μTBS test for all tested groups. According to stereomicroscope analysis, the predominant failure mode was mixed failure for most of the tested groups, except Group TSC. The highest rate of mixed failure mode was observed in Group TSC+OBU and Group SB+OBU. The second most frequent failure type is adhesive

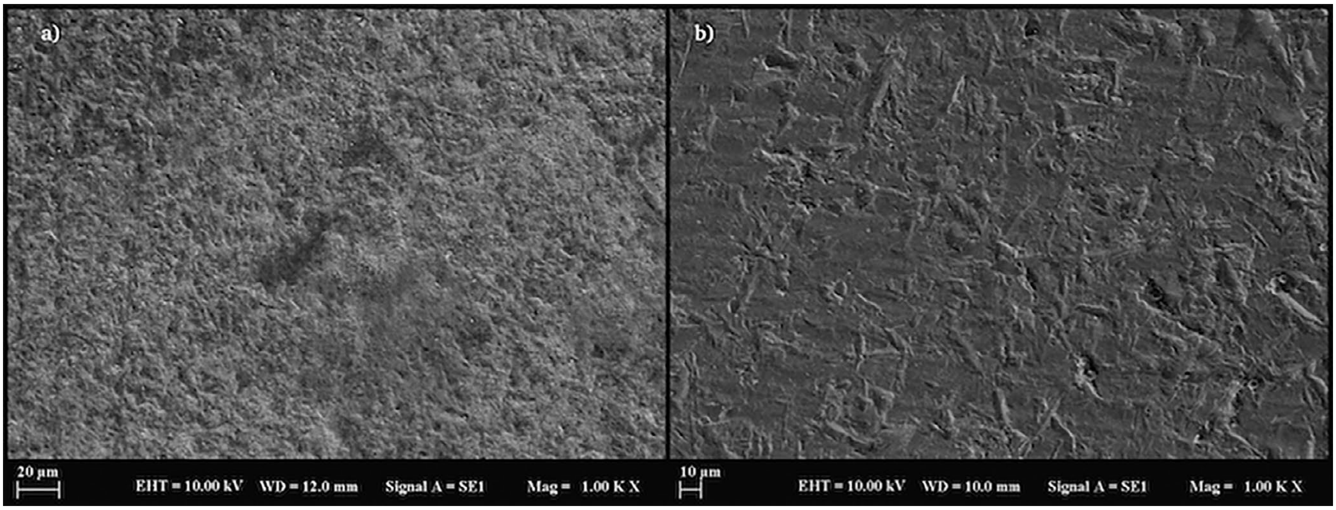

FIGURE 2 | Representative SEM images of zirconia-based CAD/CAM blocks surfaces pretrated by tribochemical silica coating (a) and sandblasting (b) (1000x).

failure, which occurs between zirconia and luting cement, and it was predominantly observed in Group TSC. The other type of failure was cohesive failure and was most frequently seen in Group SB+CUQ. Group SB exhibited cohesive failure and adhesive failure in zirconia and luting cement equally.

4 | Discussion

In the present study, the effects of IDS using different universal adhesives on μTBS of pretreated monolithic translucent tetragonal polycrystalline zirconia-based CAD/CAM restoration to dentin and microscopic morphological alterations were assessed.

In terms of results, the null hypothesis proposed that IDS application using different universal adhesive systems would not affect μTBS of pretreated monolithic translucent tetragonal polycrystalline zirconia-based CAD/CAM restoration to dentin and microscopic morphological alterations was partially rejected since after both surface pretreatment methods, the μTBS to dentin of monolithic zirconia-based CAD/CAM material was found to be higher for IDS with Optibond Universal adhesive, compared to IDS with Clearfil Quick Bond.

With the penetration of resin monomers into the hard tissue, an "interphase layer" called the hybrid layer is formed. With IDS application, a resin–dentin hybridization layer similar to the enamel–dentin connection is provided. This method, also known as "pre-hybridization", "dual bonding", and "resin coating" technique (Helvey 2011). Adhesive system selection is an important factor in the success of IDS application. In this study, for IDS, the new one-step self-etch adhesives known as "universal adhesive systems" containing different monomers (glycerophosphate dimethacrylate/GPDM, hydroxylethyl methacrylate/HEMA, 10-methacrylooxysidecyl dihydrogen phosphate/10-MDP, amide monomers) were used with self-etch mode since this mode could provide high penetration to dental tissues and reduce post-operative sensitivity compared to etch&rinse systems (Günsel, Atalı, and Türkmen 2020).

The surface roughness and morphology of the two bonding surfaces have a significant influence on the micromechanical interlocking of the interface. Before cementation of zirconia-based ceramics, sandblasting (SB) with Al_2O_3 particles is widely used as a surface pretreatment method, providing micromechanical interlocking. Tribochemical silica coating (TSC), such as with the Rocatec or CoJet systems, is a frequently utilized commercial method in which the zirconia surface is pretreated with alumina particles that have been coated with nano-silica. This causes the nano-silica to become embedded in the zirconia surface. It could enhance the adhesion capacity by utilizing a combination of surface roughening and chemical bonding (Comino-Garayoa et al. 2021).

Self-adhesive resin cements accomplish the technique sensitivity of adhesive luting systems and simplify clinical procedures since no pretreatment of the tooth surface is needed (Monticelli et al. 2008). (Oyagüe et al. 2009) indicated that self-adhesive resin cements were suitable for the cementation of zirconia-based ceramics. The previous studies indicated that higher bond strength of zirconia-based ceramics was obtained with self-adhesive resin cements compared to adhesive resin cements (Lee et al. 2019; Peçanha et al. 2022). Thus, in the present study, a self-adhesive dual-cure resin cement, RelyX U200 was preferred for the cementation of monolithic zirconia-based CAD/CAM blocks.

Silanes have been utilized to enhance the adhesion between resin materials and ceramics containing a glassy matrix (Scaminaci Russo et al. 2019). Some studies reported that similar effects on enhancing zirconia-resin bond strengths were achieved with TSC+silanization and regular air abrasion with alumina particles, thus indicating TSC only provided air–abrasion effect for creating surface roughness (Erdem et al. 2014). However, the application of silane did not improve bond strength when exposed to oral environment (Comino-Garayoa et al. 2021; Erdem et al. 2014). Thus, in this study, silanes were not used according to the manufacturer's instructions since monolithic zirconia-based materials does not contain glassy matrix. Besides, it remains unclear whether this is caused by the silica coating or by the roughening effect caused by air abrasion.

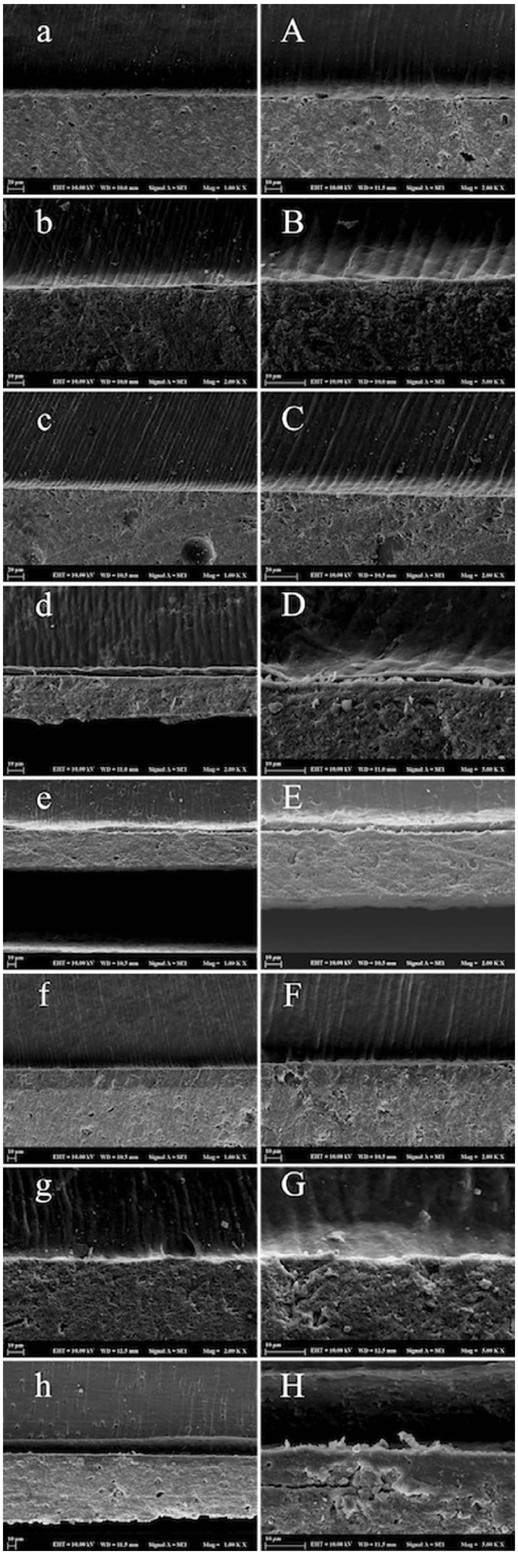

FIGURE 3 | Representative SEM images of the zirconia–dentin interfaces of all tested groups (1000×, 2000×, and 5000×). Group TSC, tribochemical silica coating+No IDS (a, A); Group TSC+SBU, tribochemical silica coating+Single Bond Universal (b, B); Group TSC+CUQ, tribochemical silica coating+Clearfil Universal Bond Quick (c, C); Group TSC+OBU, tribochemical silica coating+Optibond Universal (d, D); Group SB, sandblasting+No IDS (e, E); Group SB+SBU, sandblasting+Single Bond Universal (f, F); Group SB+CUQ, sandblasting+-Clearfil Universal Bond Quick (g, G); Group SB+OBU, sandblasting+Optibond Universal (h, H).

Thermocycling is an aging method using distilled water that exposes specimens to the temperature changes that occur in the oral cavity as a result of the consumption of hot or cold beverages (Scaminaci Russo et al. 2019). In the current study, all specimens underwent 10.000 thermocycles (5°C–55°C), which is equivalent to one year of clinical use (Morresi et al. 2014).

(Sailer et al. 2012), who evaluated the effect of IDS on the dentin bond strength of various resin cements, found that IDS application increased the dentin bond strength of a self-adhesive resin cement, RelyX U200 since IDS application using adhesive systems allows preliminary preparation of dentin. Previous studies highlighted that IDS application could improve the bond strength of resin cements to dentin (Rigos et al. 2019; Duarte et al. 2009). On the other hand, (Deniz et al. 2021) indicated that no significant effects in early bond strength of adhesive luting cement to dentin were found for IDS application with Single Bond Universal compared to no IDS application. In the present study, according to the presence of IDS, for TSC pretreated groups, IDS application with Optibond Universal caused significantly higher μTBS than no IDS application whereas, for SB pretreated groups, no significant differences in μTBS were found between IDS application and no IDS application. The highest rate of adhesive failures in stereomicroscope analysis and gap formations in the hybrid layer at the tooth–restoration interface in SEM images could confirm the lower bond strength for TSC pretreated group with no IDS.

Universal adhesive systems may exhibit different clinical performances because they have specific functional monomers containing acidic groups and interact quite differently with hydroxyapatite of tooth tissue, thus influence their bonding efficacy (Wang et al. 2017). In this study, when the IDS applied groups were compared, for both surface pretreatments, IDS with Optibond Universal resulted in significantly higher μTBS than the one with Clearfil Universal Bond Quick. As functional monomers, Optibond Universal contains GPDM while Clearfil Universal Bond Quick has amide monomer and 10-MDP. The hydrophilicity of GPDM is greater than that of MDP; as a result, GPDM has a higher wettability and is able to penetrate deep into the dentin and form a strong micro-mechanical bond (Wang et al. 2017). In addition, the molecular structure of GPDM contains two polymerizable groups, compared to MDP with only one polymerizable group. Therefore, a higher degree of polymerization is associated with an improved quality of the polymer network (Wang et al. 2017; Yoshihara et al. 2018). The lower pH acidity of Optibond Universal (pH = 1.9) could provide more aggressive etching than Clearfil Universal Bond Quick (pH = 2.3). According to the SEM images at the tooth–restoration interface, the higher bond strength of Optibond Universal could be attributed to the strong micromechanical interlocking resulting from thick hybrid layer with longer resin tags.

Besides, stereomicroscope observations confirmed this finding that highest frequency of mixed failure, in addition to cohesive failures for IDS with Optibond Universal, since (Al-Salehi and Burke 1997) indicated that increased in cohesive and mixed failures correlated with improved bond strength.

In the current study, for both surface pretreatments, IDS with Optibond Universal exhibited similar μTBS to the ones with Single Bond Universal. Single Bond Universal contains 10-MDP

TABLE 4 | Failure mode analysis of fractured surfaces after μTBS test for all tested groups (%).

Groups	Zirconia-cement adhesive	Dentin-cement adhesive	Cohesive	Mixed	Total
Group TSC	**9**	0	0	7	**16**
Group TSC+SBU	4	0	2	**10**	**16**
Group TSC+CUQ	4	0	1	**11**	**16**
Group TSC+OBU	0	0	1	**15**	**16**
Group SB	1	0	1	**14**	**16**
Group SB+SBU	2	0	1	**13**	**16**
Group SB+CUQ	4	0	3	**12**	**16**
Group SB+OBU	0	0	1	**15**	**16**

Abbreviations: Group SB, sandblasting+no IDS; Group SB+CUQ, sandblasting+Clearfil Universal Bond Quick; Group SB+OBU, sandblasting+Optibond Universal; Group SB+SBU, sandblasting+Single Bond Universal; Group TSC, tribochemical silica coating+no IDS; Group TSC+CUQ, tribochemical silica coating+Clearfil Universal Bond Quick; Group TSC+OBU, tribochemical silica coating+Optibond Universal; Group TSC+SBU, tribochemical silica coating+Single Bond Universal.

and HEMA monomers, Vitrebond (polyalkenoic acid) copolymer. HEMA is often added to improve dentin wetting and increase bond strength (Yoshida, Yoshihara, and Hayakawa, et al. 2012). However, the previous studies have reported that 10-MDP monomer can chemically bond with hydroxyapatite crystals and form a nanolayer at the bonding interface (Yoshida, Yoshihara, and Nagaoka, et al. 2012). The Vitrebond copolymer bonds chemically to the calcium in hydroxyapatite (Perdigão, Sezinando, and Monteiro 2012). Several studies found that the bond strength of this universal adhesive was enhanced by Vitrebond copolymer, which promoted chemical bonds in these nanolayering structures (Perdigão, Sezinando, and Monteiro 2012; Yoshida et al. 2000). Therefore, this finding could be attributed to the additional bond strength of Vitrebond copolymer for Single Bond Universal.

In the present study, for both surface pretreatments, no significant differences were detected in μTBS between Clearfil Universal Bond Quick and Single Bond Universal. The multifunctional amide monomer of Clearfil Universal Bond Quick has higher hydrophilic properties than HEMA monomer and shows higher wettability in a short time (Kuno et al. 2019). However, it was reported that the reduced application time of this adhesive system resulted in insufficient solvent evaporation and inferior bonding performance. It was demonstrated that higher bond strength was achieved when the application time of Clearfil Universal Bond Quick was extended to 20s. Thus, in this study, it was decided that all universal adhesive systems were applied for 20s since the application procedure was intended to be similar for all adhesives (Ahmed et al. 2020). In the current study, the bond strength results are supported by tooth-restoration interface and failure mode analyses for Clearfil Universal Bond Quick and Single Bond Universal. SEM analysis revealed that thin and continuous hybrid layer with short resin tags were observed. Besides, they exhibited mixed failures in addition to adhesive failures.

In this study, when the surface pretreatment methods were compared, no significant differences in μTBS and surface roughness were detected for all tested groups. This finding is line with previous studies (Akyil, Uzun, and Bayindir 2010; Attia 2011), who examined the effect of different surface pretreatments on the bond strength of resin cement to zirconia-based ceramics, reported that similar bond strength was obtained with both pretreatments. Besides, it has been reported that the bond strength between resin and zirconia is unstable despite the application of TSC. This may be due to the weak attachment of silica to zirconia surfaces (Inokoshi et al. 2014).

On the other hand, (Atsu et al. 2006), who examined the effect of different surface pretreatments on the bond strength of adhesive resin cement to zirconia-based ceramics, reported that SB (125 μm Al_2O_3) caused lower bond strength than TSC. The divergence in outcomes of this study and Atsu et al.'s study could be explained by the differences in type of luting cements (self-adhesive and adhesive resin cements) and the particle size (50 and 125 μm) of Al_2O_3. Moreover, (Hayran, Kuşçu, and Sarıkaya 2021), who investigated the effect of different surface pretreatments on the bond strength of resin cement to zirconia-based ceramics, reported that SB application to zirconia surface provides higher shear bond strength than TSC. The divergence in outcomes of this study and Hayran et al.'s study could be explained by the differences in type of particle size of silica (50 μm) for TSC and type of bond strength test method (shear bond strength).

5 | Conclusions

This in vitro study had some limitations. In this study, no cavity preparations were performed. In future studies, it would be more accurate to evaluate the contribution of IDS application on microtensile bond strength after various cavity preparations for monolithic translucent zirconia-based CAD/CAM restorations.

Within the limitations of this in vitro study, it can be concluded that:

1. After both surface pretreatments of monolithic zirconia, IDS procedure with Optibond Universal could affect the bond strength to dentin and morphological alterations, positively. IDS with Optibond Universal resulted in higher bond strength than the ones with Clearfil Universal Bond Quick. Besides, SEM images of Optibond Universal revealed that thick hybrid layer with longer resin tags was observed.

2. When the surface pretreatment methods were compared, no significant differences in μTBS and surface roughness were detected for all tested groups.

Author Contributions

Leyla Fazlioglu: resources, writing – original draft, investigation, formal analysis, writing – review and editing, funding acquisition, data curation. **Burcu Oglakci ozkoc:** writing – review and editing, project administration, software, supervision, visualization, funding acquisition, methodology. **Dilek Tagtekin:** resources, methodology.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

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